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Patrick Ruth et al. (PRSM), "Effects on Processing by Drop-in Modifiers in Nano-Composite Polymers"

**SAMPE Industry Conference** **(Statement A)**
**(Long Beach, CA, 12-15 May 2002)** **(Deadline: 12 May 2002)**

# *"Effects on Processing by Drop-in Modifiers in Nano-Composite Polymers "*


**Patrick Ruth,**
Senior Technician, AFRL/PRSM
Air Force Research Lab, Edwards
**Brent Viers, Rusty Blanski, and Andre Lee**

# POSS as a Drop-in Modifier- Introduction

What is POSS? (Simplified)

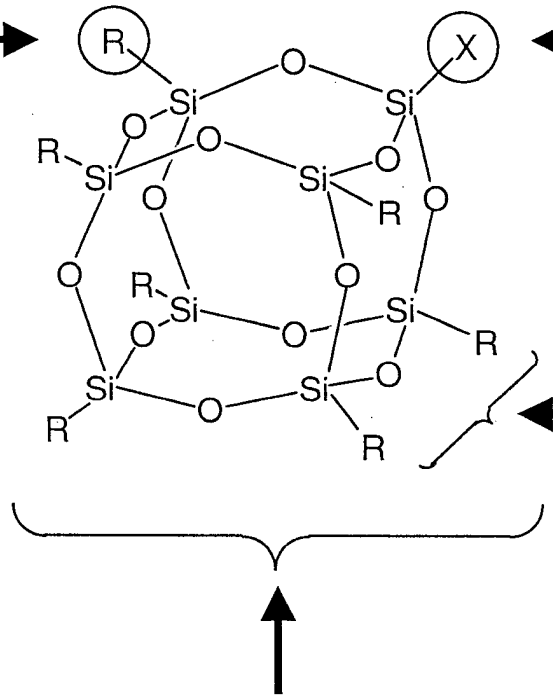
1. Structure
2. Functional Groups and Dropping-in
3. Proposed and Actual uses

Making Samples

1. Material Selection and Preparation
2. Blending
3. Sample Production

# Anatomy of a Polyhedral Oligomeric Silsesquioxane (POSS™) Molecule

Nonreactive organic (R) groups for solubilization and compatibilization.



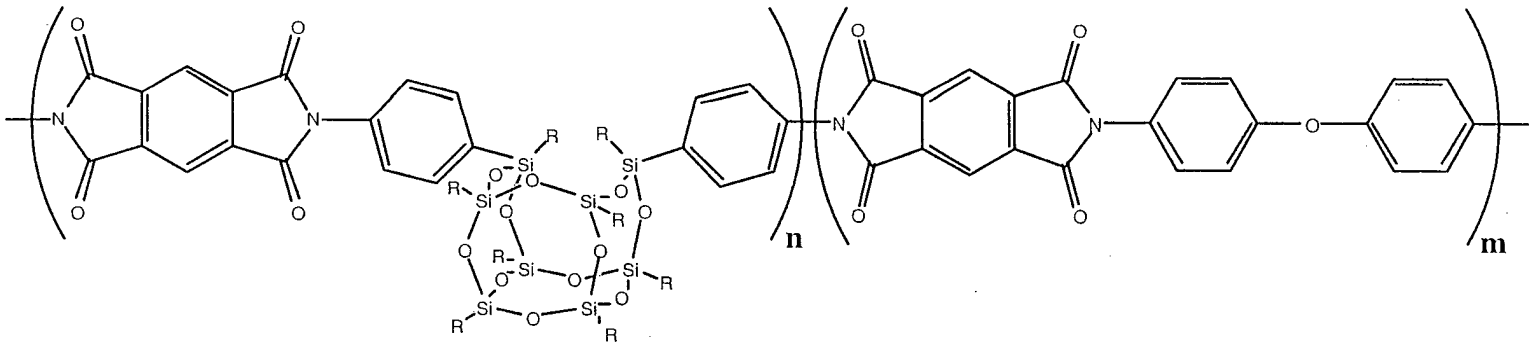

May possess one or more functional groups suitable for polymerization or grafting.

Nanoscopic in size with an Si-Si distance of 0.5 nm and a R-R distance of 1.5 nm.

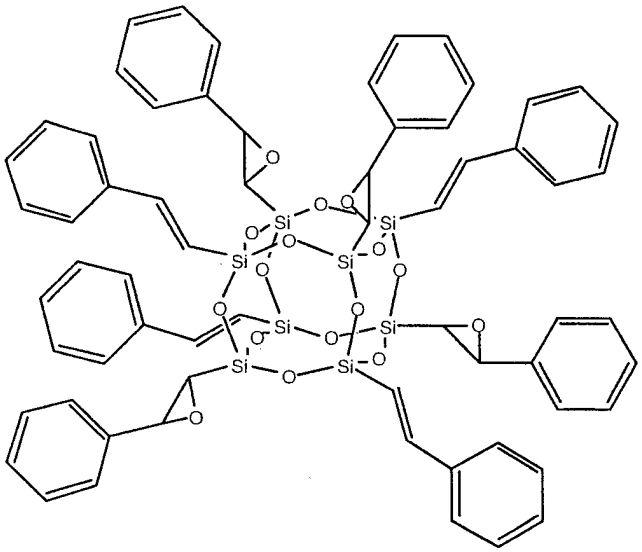
Thermally and chemically robust hybrid (organic-inorganic) framework.

Precise three-dimensional structure for molecular level reinforcement of polymer segments and coils.

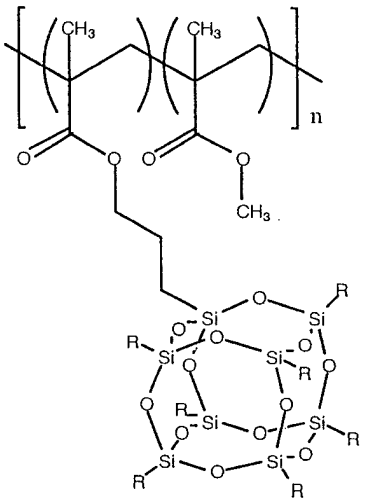
# POSS Chemically Incorporated into Plastics



POSS-Kapton

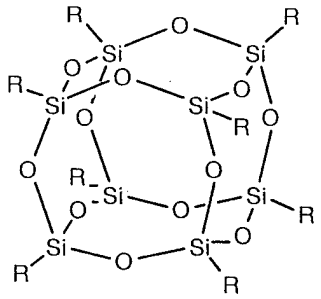


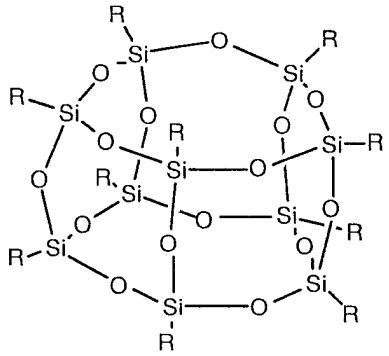
POSS-EPOXY

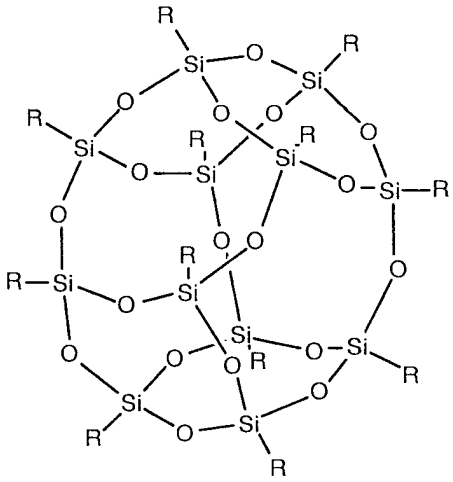


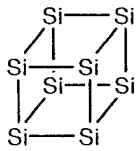
POSS-PMMA

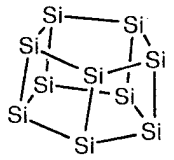
# POSS Blended into Plastics

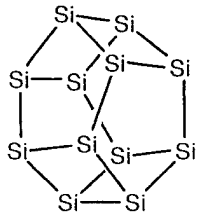




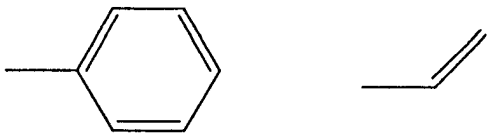




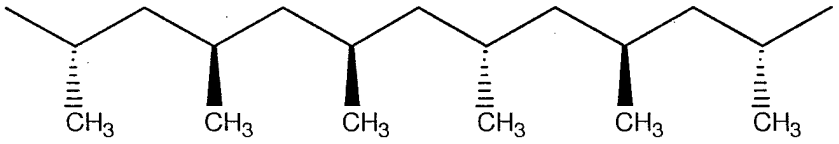




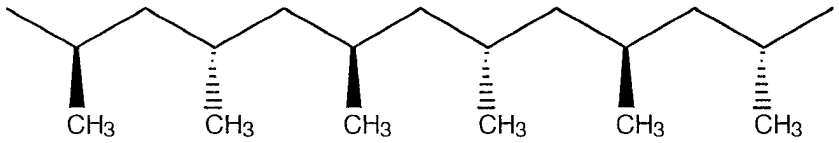
$T_8$ $T_{10}$ $T_{12}$

R = 

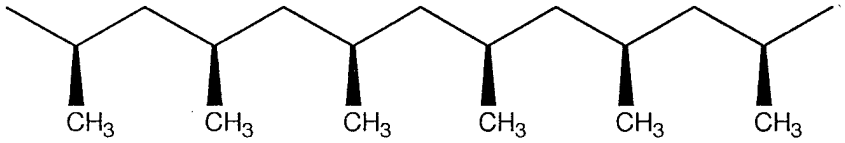
# Materials Selection: Polypropylene and POSS



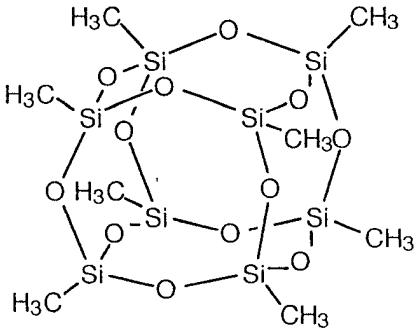

atactic polypropylene




syndiotactic polypropylene

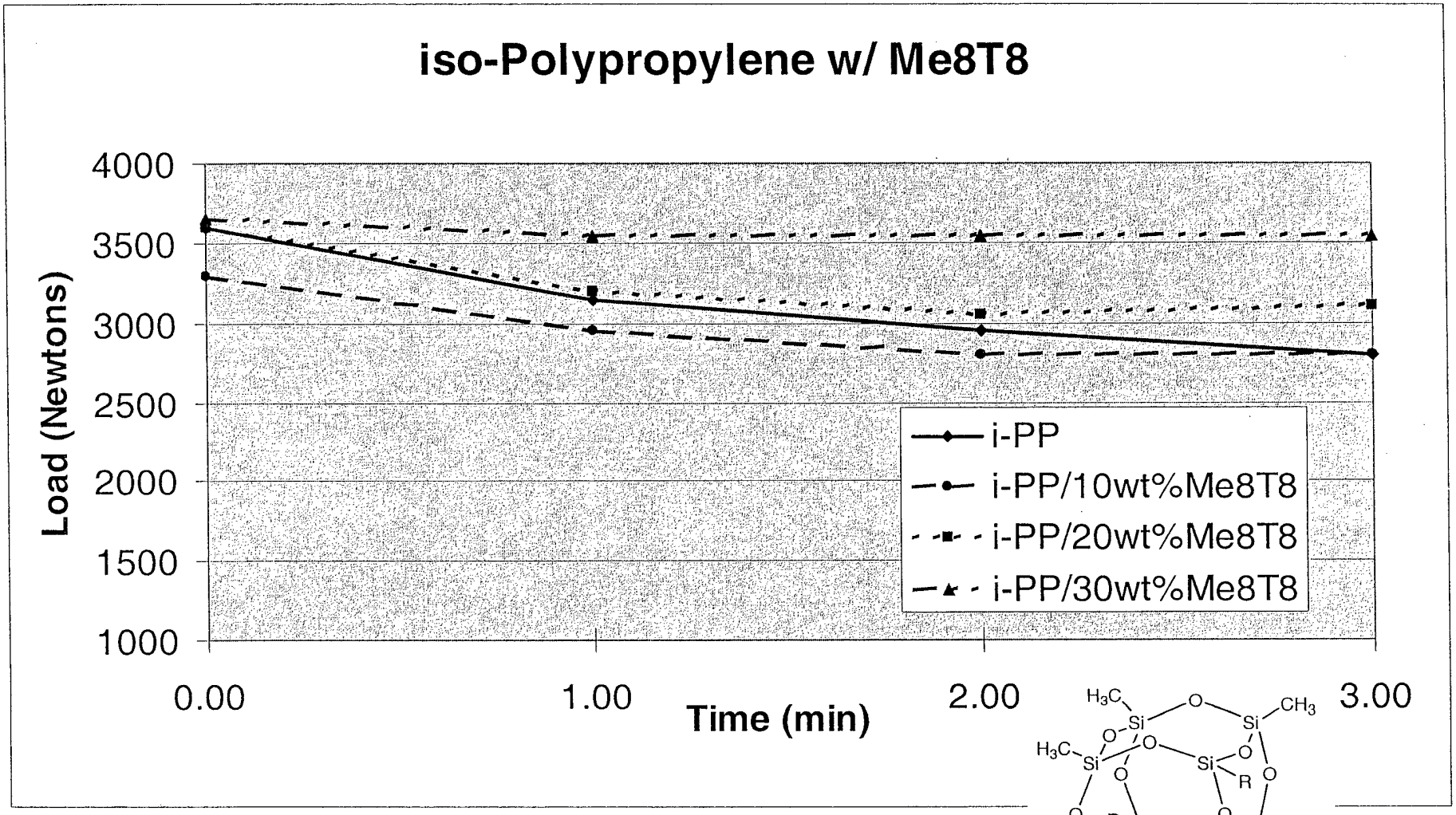



isotactic Polypropylene




$Methyl_8T_8$

# *i-PP/$Me_8T_8$ Processing Studies*

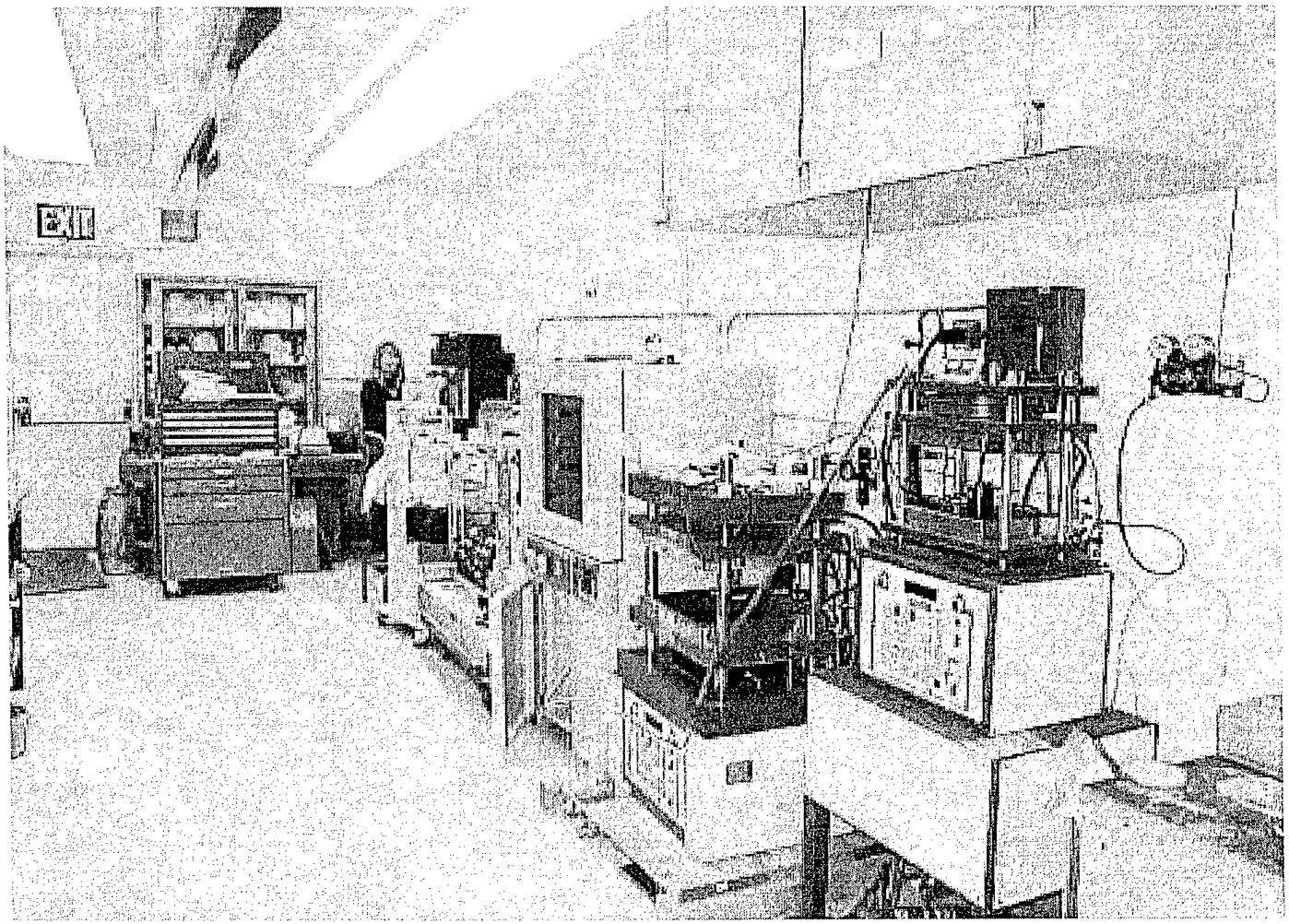


# *Prof. Andre Lee - Michigan State University*

| | Dow data | Neat *i*-PP (processed) | *i*-PP blended 2 wt% $Methyl_8T_8$ | *i*-PP blended 5 wt% $Methyl_8T_8$ | *i*-PP blended 10 wt% $Methyl_8T_8$ |
|---|---|---|---|---|---|
| Tensile Strength @ Yield; ASTM D638 | 5000 psi (34.5 MPa) | 4800 psi (33.0 MPa) | 5000 psi (34.5 MPa) | 5100 psi (35.1 MPa) | 5200 psi (35.8 MPa) |
| Flexural Modulus (0.05 in/min, 1% secant); ASTM D790A | 240,000 psi (1.655 GPa) | 235,000 psi (1.620 GPa) | 251,000 psi (1.730 GPa) | 255,000 psi (1.757 GPa) | 262,000 psi (1.80 GPa) |
| HDT @ 66 psi, as injected; ASTM D648 | 210 °F (99 °C) | 210 °F (99 °C) | 221 °F (105 °C) | 239 °F (115 °C) | 255 °F (124 °C) |
| Impact Izod @25C ASTM D256A | 0.5 ft-lb/in | 0.55 ft-lb/in | 0.55 ft-lb/in | 0.62 ft-lb/in | 0.75 ft-lb/in |

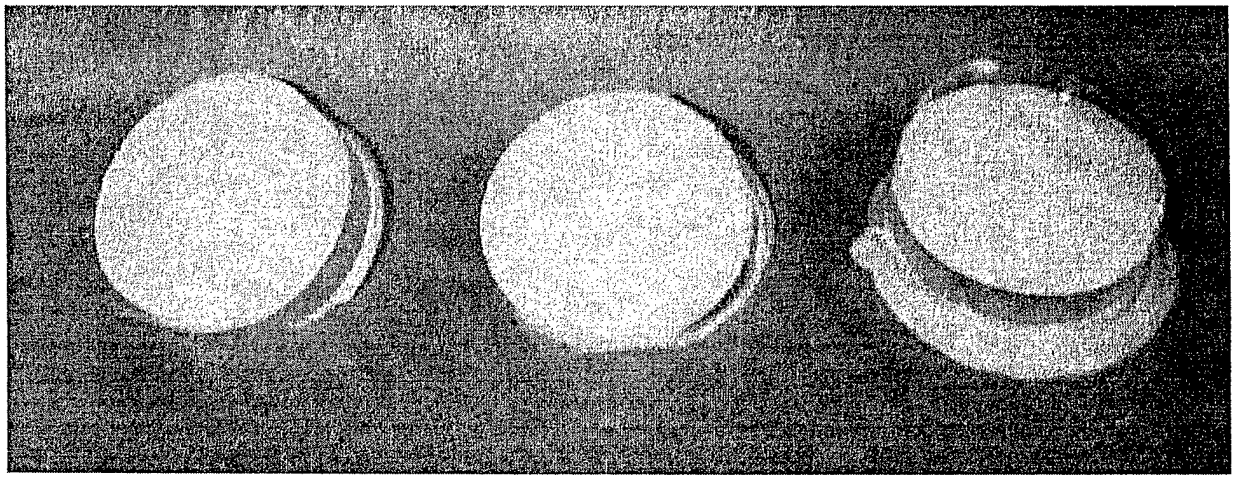
- **The above data (other than Dow's data) is an average of at least 10 samples for each test with acceptable S.D. of 5% or better.**

# Polymer Processing Lab



# Polymer Processing Parameters

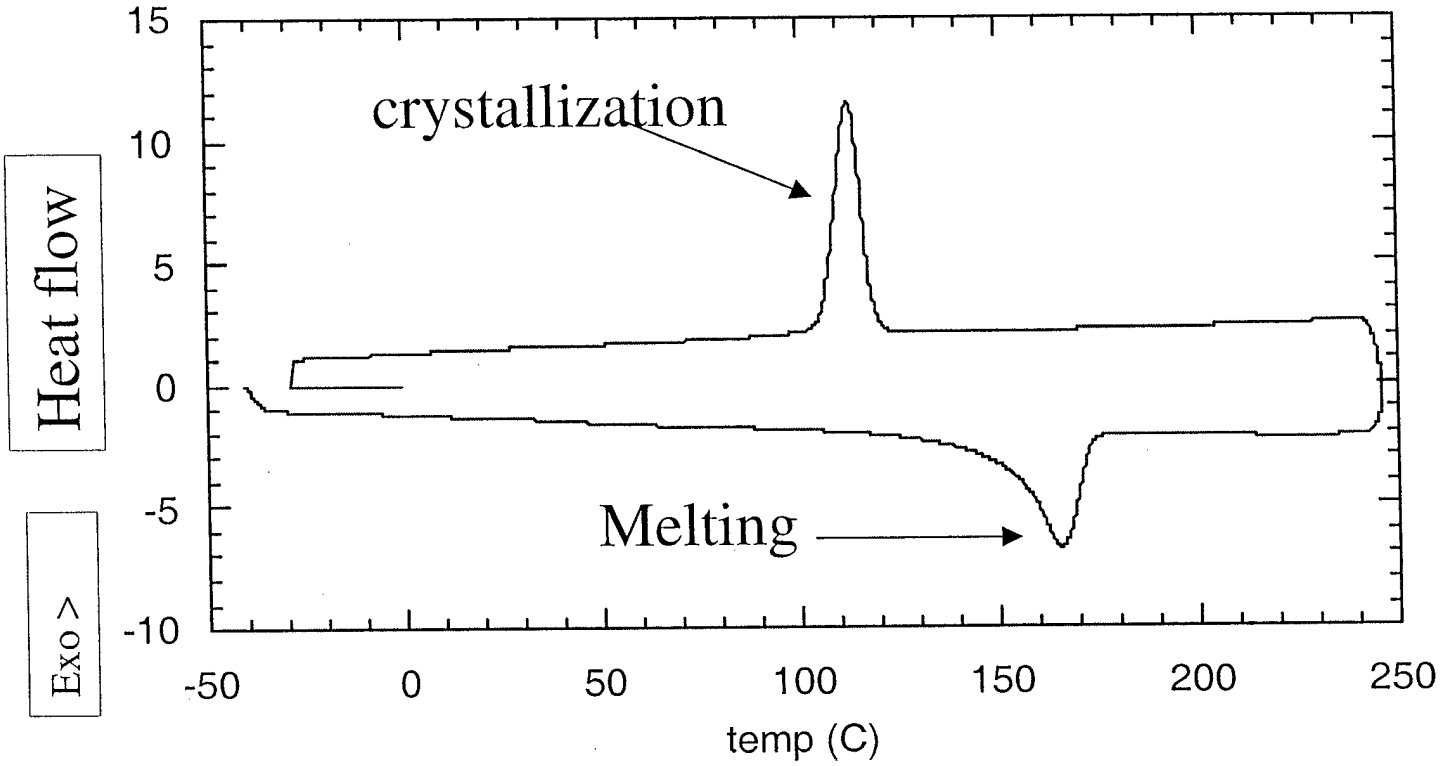
- Time
- Pressure
- Temperature



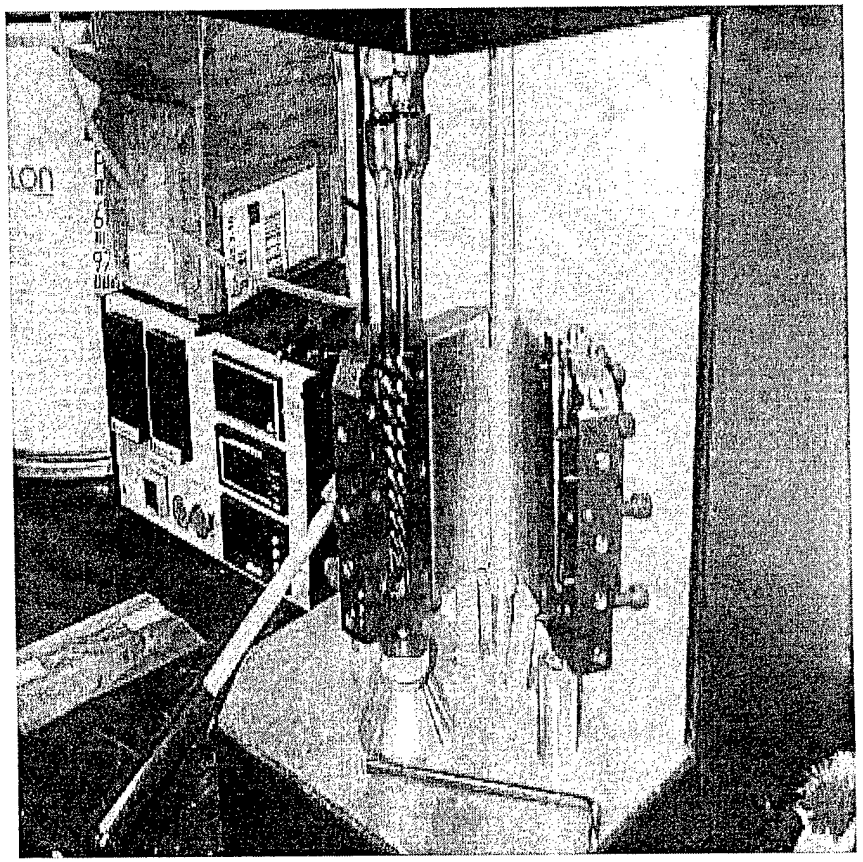
# Procedure

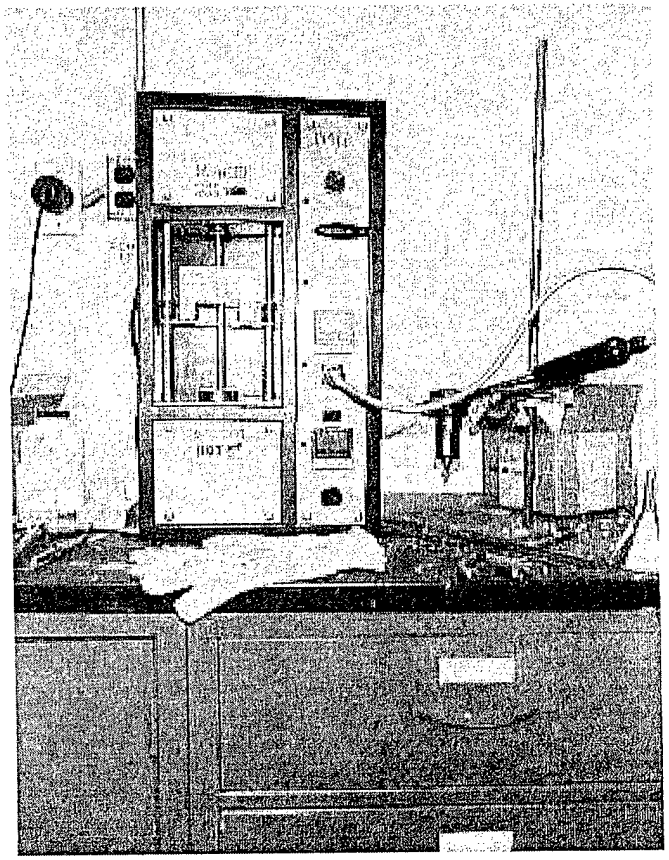
- DSC (Establish processing and drying temperatures)
- Drying (Vacuum Oven)
- DACA (Mixing)
- Press (Forming samples)
- Tests to compare properties

# Polypropylene DSC



# DACA

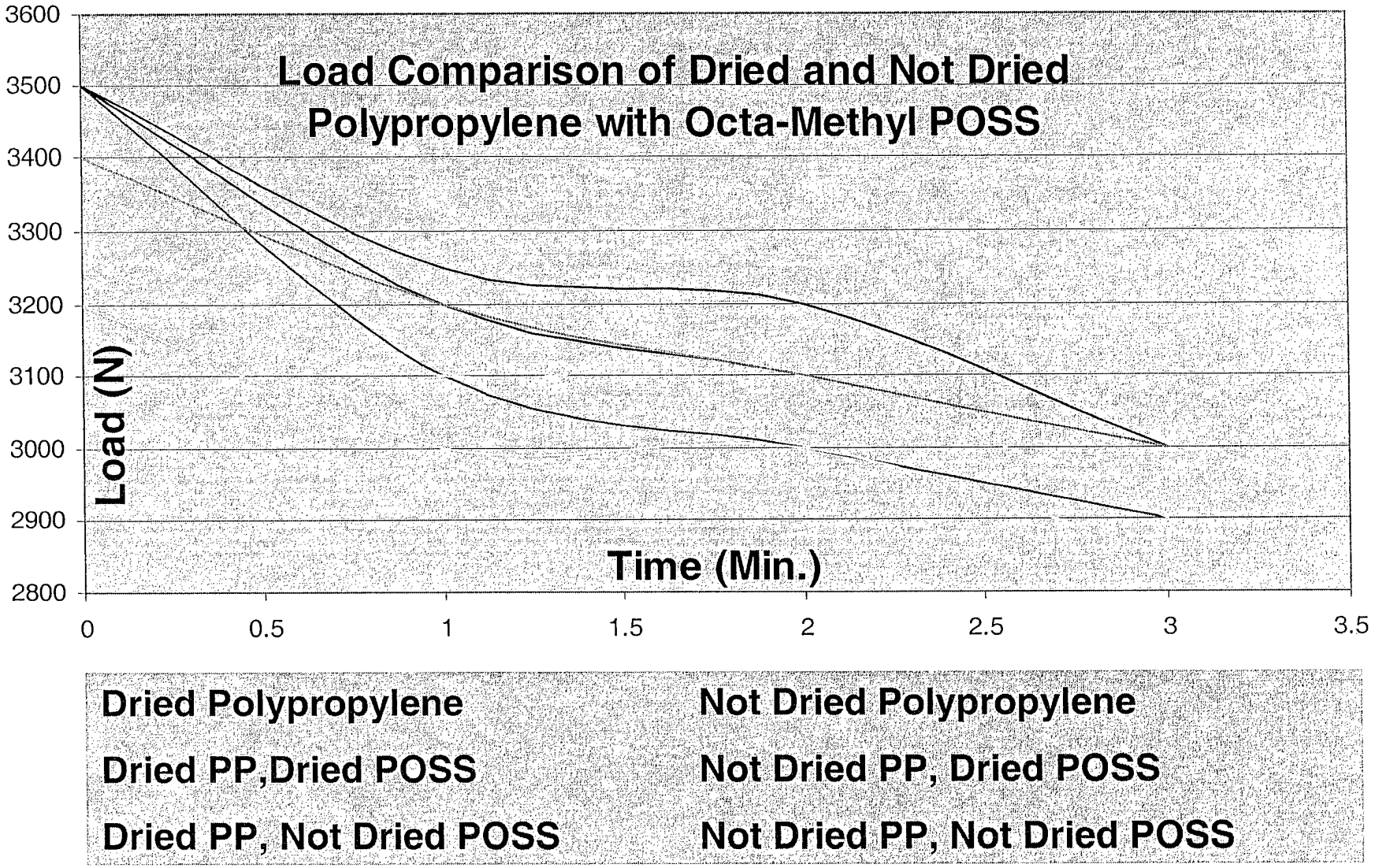


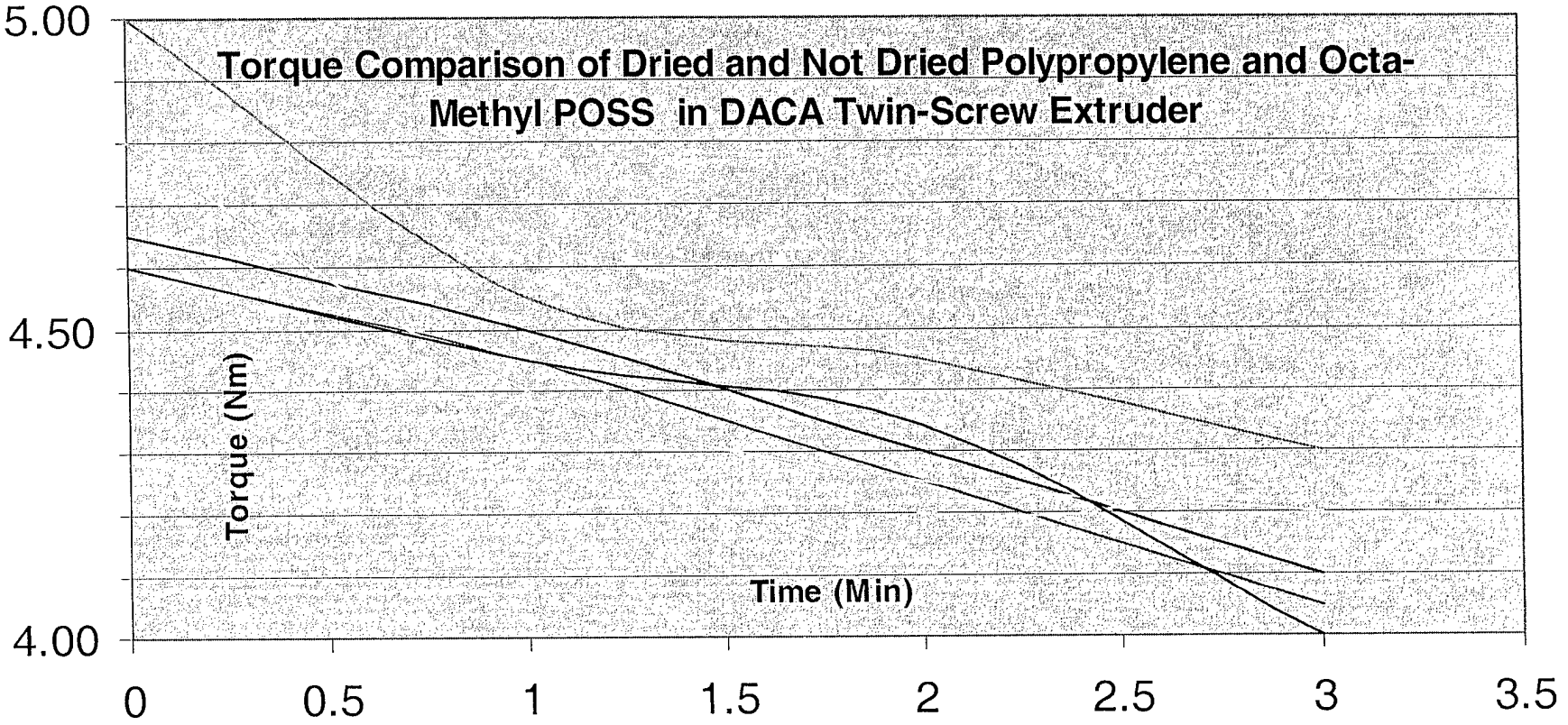


# DACA Twin Screw Processing Parameters for Me8T8/iPP nanocomposite blends.

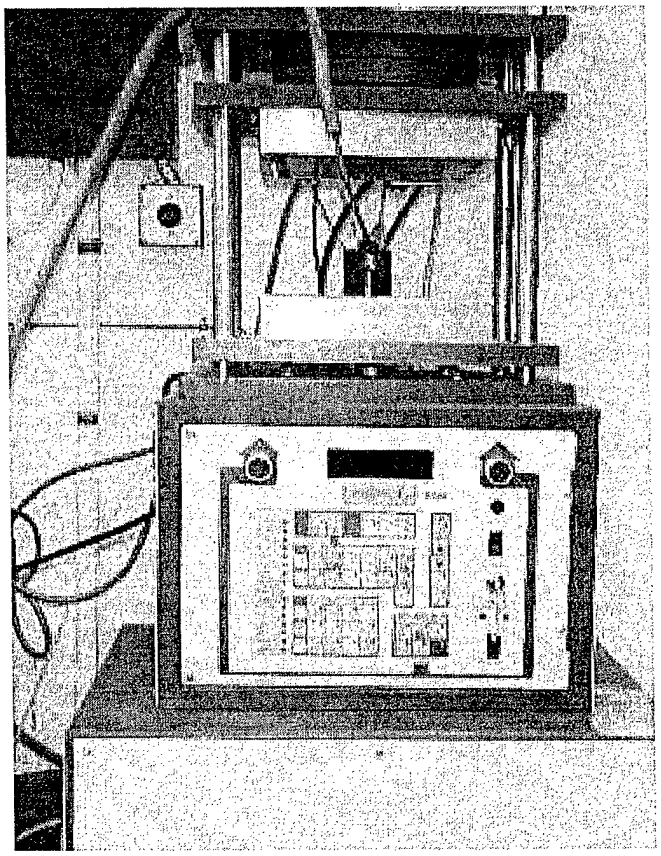
| Mix # | Material Percentage | | | | Load (N) | | | | Torque (Nm) | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | PP | | $Me_8T_8$ | | Mix Duration (min) | | | | Mix Duration (Min) | | | |
| | Dried | Not Dried | Dried | Not Dried | 0 | 1 | 2 | 3 | 0 | 1 | 2 | 3 |
| 1 | | 100 | | | 3500 | 3200 | 3100 | 3000 | 4.65 | 4.50 | 4.30 | 4.10 |
| 2 | 100 | | | | 3500 | 3100 | 3000 | 2900 | 4.60 | 4.45 | 4.25 | 4.05 |
| 3 | 90 | | 10 | | 3200 | 3000 | 3000 | 2850 | 4.80 | 4.40 | 4.25 | 4.20 |
| 4 | 90 | | | 10 | 3200 | 3100 | 3100 | 2900 | 4.60 | 4.45 | 4.20 | 4.25 |
| 5 | 90 | | 10 | | 3500 | 3250 | 3200 | 3000 | 5.00 | 4.55 | 4.45 | 4.30 |
| 6 | | 90 | | 10 | 3400 | 3200 | 3100 | 3000 | 4.60 | 4.45 | 4.34 | 4.00 |

Hot Press


Load Comparison of Dried and Not Dried Polypropylene with Octa-Methyl POSS
Load (N)
Time (Min.)
3600
3500
3400
3300
3200
3100
3000
2900
2800
0
0.5
1
1.5
2
2.5
3
3.5
Dried Polypropylene
Dried PP,Dried POSS
Dried PP, Not Dried POSS
Not Dried Polypropylene
Not Dried PP, Dried POSS
Not Dried PP, Not Dried POSS

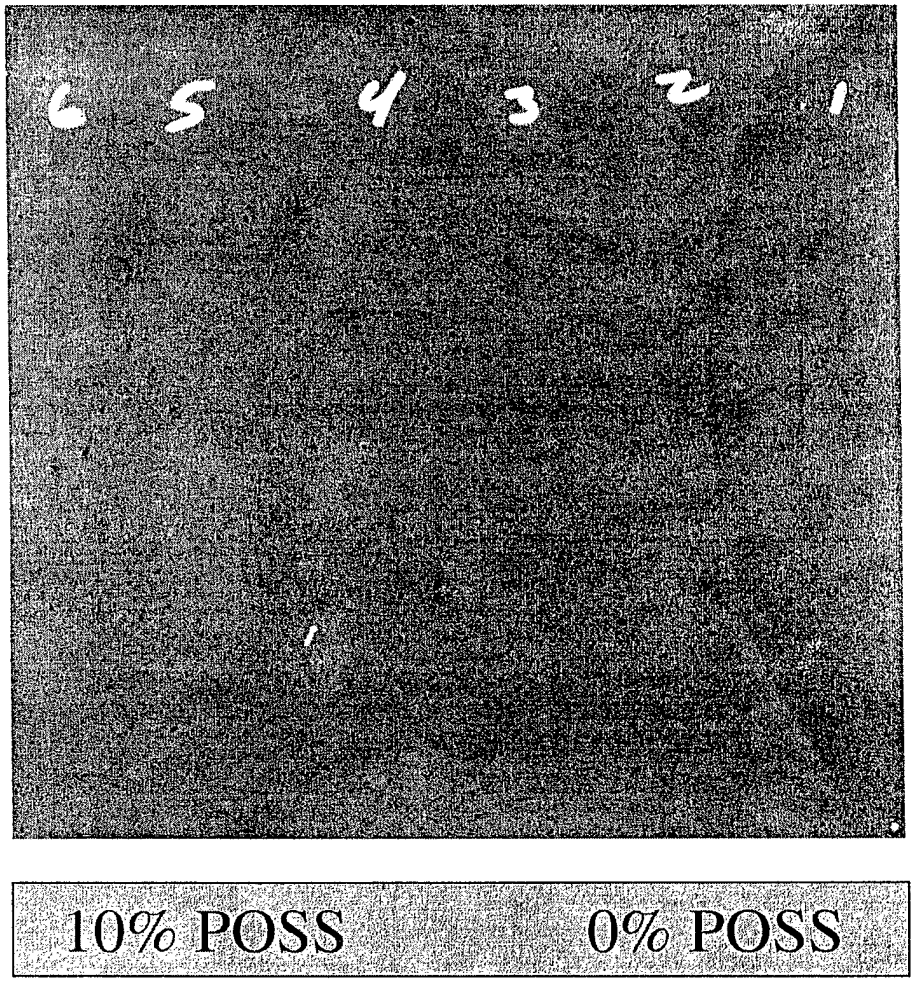

Torque Comparison of Dried and Not Dried Polypropylene and Octa-Methyl POSS in DACA Twin-Screw Extruder
5.00
4.50
4.00
Torque (Nm)
Time (Min)
0
0.5
1
1.5
2
2.5
3
3.5
Dried Polypropylene
Not Dried Polypropylene
Dried PP,Dried POSS
Not Dried PP, Dried POSS
Dried PP, Not Dried POSS
Not Dried PP, Not Dried POSS

# One Ton Press



# Pressed film of DACA extruded POSS/PP blend variants

1 Dried PP
2 Not Dried PP
3 Dried PP, Dried POSS
4 Dried PP, Not Dried POSS
5 Not Dried PP, Dried POSS
6 Not Dried PP, Not Dried POSS



# *SUMMARY*

**Drying seems to play a roll in making $Me_8T_8$ compatible with isotactic polypropylene**

**Load/torque to mix the polymer with the POSS is increased if either of the components is not dried.**

**Visually, the most compatible of the mixes is number 3 where both POSS and PP components were dried. The extruded rod and pressed thin film are nearly as clear as pure polypropylene in the melt.**

# *ACKNOWLEDGEMENTS*

**AFRL/PRSM: Dr. Brent Viers, Dr. Rusty Blanski, and Dr. Andre Lee**
**Air Force Research Lab Polymer Working Group**

**Hybrid Plastics: Dr. Joe Lichtenhan, Dr. Joe Schwab, and**
**Mr. Michael J Carr**

**This talk is as much about me learning my work as it is making samples. A great deal of thanks goes to the people who do similar work and have shown me tricks to make the technician look clever.**